



Study of the effectiveness of quick tests based on physical properties for the evaluation of used frying oil

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ABSTRACT

The aim of this paper was to evaluate the effectiveness of the quick tests Viscofrit, Testo 265 and Fri-check in the monitoring of used frying oil for discarding, as a substitute for the measurement of total polar compounds (TPC), indicating the advantages and disadvantages of each test. Fifty-nine frying oil samples used to fry different types of food were evaluated using quick tests and the TPC open-column conventional method. The samples presented TPC values from $6.0 \pm 1.1\%$ to $39.2 \pm 1.0\%$. Testo 265 was correlated to the conventional method by the equation: $y = 0.89x + 4.0$ ($r = 0.90$), with a 5% level of significance. A correlation factor of 0.9 ($\alpha = 0.05$) was essential for these results, since water in the food increases the TPC values. Viscofrit provided results consistent with the open-column method for 78.8% of the samples, with 9.3% of false positive results. Testo 265 and Viscofrit showed good results for the substitution of the TPC conventional method, respecting their limitations. Viscofrit is not recommended for high melting point samples.

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1. Introduction

The deep-fat frying of food is an important option since it is a quick preparation process and also provides characteristic smells and flavors to the products (Varela, Bender, & Morton, 1988). A set of complex changes and reactions producing several degradation compounds, occurs during this operation (Fritsch, 1981).

Throughout the frying process, the oil or fat is exposed to high temperatures, ranging from 162 to 196 °C, in the presence of air and water (Dobarganes & Márquez-Ruiz, 1998; Sanibal & Mancini Filho, 2002; Varela et al., 1988). Under these conditions, a set of simultaneous reactions occurs, such as thermoxidation, polymerization and hydrolysis, resulting in quality losses of both frying oil and food (Dobarganes & Márquez-Ruiz, 1998; Saguy & Dana, 2003). The improper monitoring of frying oil quality can cause both risks to public health and economic prejudice to restaurants (Paul & Mittal, 1997).

No method is capable of demonstrating the changes in used frying oil throughout the whole process. It is worth recalling that the available methods provide information on particular stages of

the degradation process. Moreover, the method of choice will depend on the type of information desired, the time availability, and also the conditions required for the test.

On one hand, some food frying systems present high water contents and minimal oxidative conditions, requiring tests which reflect hydrolytic changes, such as the acidity and smoke levels, for instance. On the other hand, systems which favor oxidation, such as those containing highly unsaturated oil and applying high temperatures, will require tests that reflect the oxidative state (Lima & Gonçalves, 1995). One of the commonest tests is the determination of the total polar compounds (TPC).

The polar fraction of frying oils is composed of polymers and decomposition products. The polymers (dimers, trimers and highly polymerized compounds) comprise all the degradation products whose molecular weights are higher than those of the triacylglycerols. However, decomposition products (mono and diacylglycerols, free fatty acids, volatile compounds and cyclic and non cyclic monomers) are composed of degradation products whose molecular weights are lower than those of the triacylglycerols (Dobarganes, Velasco, & Dieffenbacher, 2000; Jorge & Gonçalves, 1998; Paul & Mittal, 1997).

Of the analytical procedures used to evaluate frying oils and fats, the most trustworthy and best known method is often the TPC

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determination (Dobarganes et al., 2000; Melton, Jafar, Sykes, & Trigiano, 1994). Based on international legislation, when the TPC content reaches 25%, the frying oil is considered improper for consumption (Dobarganes & Márquez-Ruiz, 1998; Paul & Mittal, 1997). However, this determination is complex, tedious, time consuming and may not be suitable for the routine control of oil and fat quality (Chu, 1991; Dobarganes & Márquez-Ruiz, 1998).

There is a great deficiency of quick tests, which are simple and direct in the prediction of oil degradation (Innawong, Mallikarjunan, & Marcy, 2004). Of the quick tests available for the evaluation of frying oil, the most famous ones are based on changes in the dielectric constant, which is connected to the level of polar compounds in the frying oil.

An ideal quick test must fulfill most of the following requirements: measurement of the % polar compounds, high precision even at low polar concentration levels, repeatability and reproducibility of the results, unbreakable equipment, easily handled, low cost, no need to calibrate with the type of oil under analysis, to be a legitimate methodology for any type of fat and/or oil, and not to be subject to interference by salts, metals, moisture or other conductive materials which may be present in the oil under analysis. Moreover, the equipment must be compact so as to be suitable for use on the assembly line “in situ”, without the need for chemical agents or cooling of the oil before making the measurements.

There are three available systems – Fri-check (Fraiss, 2009), Testo 265 (Testo, 2009) and Viscofrit (Viscofrit, 2006) – based on physical changes in the frying oil, which can be used as quick methods to evaluate indirectly whether the polar compound content has surpassed the 25% mark. The Fri-check measurements are based on changes in the oil density, viscosity and interfacial tension. The method is reliable and the results correlate well with those obtained by the official TPC determination (Fraiss, 2009; Gertz, 2000; Osawa, Gonçalves, & Grimaldi, 2005). The Testo 265 measurements are based on changes in the dielectric constant of the frying oil. Although this test has been used repeatedly in the evaluation of frying oils (Dobarganes, 2007; Lee, Chung, Chang, & Lee, 2007; Sánchez-Gimeno, Benito, Vercet, & Oria, 2008; Sánchez-Gimeno, Negueruela, Benito, Vercet, & Oria, 2008; Zappe & Carli, 2006), conclusive studies are still necessary. The Viscofrit measurements are based on changes in the viscosity of the oil (Viscofrit, 2006).

The aim of this paper was to evaluate the effectiveness of the three quick tests (Fri-check®, Testo 265 and Viscofrit®) in the determination, directly or indirectly, of the polar compounds present in several frying oil samples, so as to be able to recommend their use to commercial establishments, sanitary surveillance agencies and domestic users.

2. Material and methods

2.1. Frying oils

The discarded frying oil samples analyzed in this study were as varied as possible. Some came from meat products coated in flour and fried in palm olein or cottonseed oil, obtained from the Uni-camp Fats and Oils Laboratory. Other samples were donated by commercial establishments that fry several types of food. In these cases, the commercial establishments donated both the used and respective fresh oils.

All the samples were kept frozen in amber bottles until analyzed. A total of 59 samples were analyzed.

2.2. Methodologies adopted

The quick tests: Testo 265 (Testo, Germany), Viscofrit (Seguridad Alimentaria Laboratory, Spain) and Fri-check (Fraiss Imp. e Exp. Ltda,

Brazil) were used to evaluate the discarded oil. Each quick test was carried out in triplicate.

The TPC open-column chromatographic method developed by Dobarganes and co-authors (Dobarganes et al., 2000) was adopted, and the determinations were carried out in duplicate. One gram of the filtered and diluted sample was dissolved in 10 mL of petroleum ether: diethyl ether 90:10 (v/v) (solution 1). An open glass chromatographic column (10 mm internal diameter, 150 mm long, polytetrafluorethylene tube with glass joints) was prepared by adding solution 1 and 5 g of silica gel with a particle size of 0.063–0.200 mm (Merck n° 7734 or equivalent), previously activated at 160 °C for at least 4 h and adjusted to 5% moisture content. The non-polar fraction was eluted with 60 mL of solution 1 at a flow rate of approximately 1.5 mL/min, and collected in a 100 mL round-bottomed flask with a ground glass neck.

The polar fraction was then eluted using ethyl ether, and collected in another 100 mL round-bottomed flask with a ground glass neck. The respective solvents used to elute the polar and non-polar fractions were evaporated off in a rotary evaporator by weight difference, and the polar and non-polar fractions quantified.

The chromatographic separation was confirmed using thin-layer chromatography, applying 2 µL of the solutions containing the non-polar matter (10% in petroleum ether or ethyl ether) to a thin-layer chromatographic plate (20 × 20 cm × 0.25 mm) with a 0.25 mm thick silica gel layer, using a capillary pipette specific for thin-layer chromatography. The mobile phase was petroleum ether: diethyl ether: acetic acid (70:40:1), and visualization was carried out using sublimed iodine at about 40 °C.

The experimental procedures used for the analyses with the quick tests are shown below.

2.2.1. Testo 265

The samples were analyzed by inserting the sensor into the previously heated (40–210 °C) oil, and reading the temperature and TPC content in percentage from the display after about 30 s. The sensor was calibrated with the calibration oil supplied by the manufacturer before analyzing the frying oils. The equipment was cleaned with warm water and neutral detergent and dried well between measurements.

2.2.2. Viscofrit

This test consists of measuring the time required in seconds for a sample of frying oil (totally liquid) to flow out of a cone-shaped funnel. The test can be applied in a temperature range of from 15 to 50 °C, but temperatures between 20 and 30 °C are recommended.

In the present study, temperatures between 21 and 39 °C were adopted for all 59 samples due to the high melting point of some of the samples, and the fact that in the range from 40 to 50 °C, there would be significant cooling of the oil during the test, contributing to inaccurate and unreliable results.

In a second trial, 19 of the samples were evaluated in two different temperature ranges, 27–39 °C and 41–50 °C, for comparative reasons, the results also being compared with those obtained using the conventional open-column method for the determination of polar compounds.

According to the type of oil (mostly monounsaturated or polyunsaturated) and the test temperature, there is a maximum predetermined period of time to empty the cone, associated with the viscosity of the oil. If the outflow time is shorter than expected, the oil is still proper for use; if it is not, then the oil has degraded and is presenting a polar compound content over 25%.

If the type of oil is unknown, the test must be performed using fresh oil, using the time and temperature established in the Viscofrit manual, in order to define the type of oil under analysis.

2.2.3. Fri-check

The device was first turned on, and reached thermal equilibrium at a temperature of 47 °C after a 5-min wait. The sample pack was then inserted and hot water added. The water was discarded after 10–20 s and the samples evaluated. The measuring tube was filled with about 15 mL of heated sample, and approximately 5 min later the sample temperature stabilized at 52 °C and the result appeared on the display as both %PM (total polar compounds) and U (Fri-check® units).

It is worth mentioning that the equipment had been calibrated by the manufacturer before the analysis. To clean the equipment, the measuring tube was washed with water and given a final rinse with ethyl alcohol, guaranteeing the absence of moisture before the measurements.

2.3. Statistical analysis

In the statistical analysis to correlate the quick tests with the conventional method and in the paired comparison tests, the first two values obtained in the quick tests were used, since the determinations of the polar compounds using the conventional method were carried out in duplicate.

For the Testo 265 and Fri-check tests, a linear correlation was made for each quick test with the results obtained by the official polar compound method, using the minimum square method (Montgomery, 1991; Montgomery & Peck, 1992) and the Minitab for Windows version 12.1 software (Minitab, 1994, 1997), with 95% of confidence bands and 95% of prediction bands (Charnet, Freire, Charnet, & Bonvino, 1999; Montgomery & Peck, 1992). The analysis of variance was carried out using the software SAS for Windows version 8.2 (Council, 1985), to validate the linear regression and for the means comparison test at the 5% significance level.

The Viscofrit results were obtained for over 25 observations (D'Hainaut, 1997) using the Signal Test at the 5% significance level, and also to determine false positive and false negative results. False positives are confirmed when the Viscofrit confirms that the polar content is >25% (YES), when in fact it is not, and false negatives are confirmed when the Viscofrit indicates that the polar content is <25% (NO), when, in fact, the oil should be discarded. In the present study, the percentage of false negatives was highly relevant, although the percentage of false positives could cause economic losses for the industry or commercial establishment, since the oil will be discarded before it has degraded.

3. Results and discussion

3.1. TPC content using the conventional method

The TPC contents of the samples obtained using the conventional method, presented values from $6.0 \pm 1.1\%$ to $39.2 \pm 1.0\%$, distributed as shown in Table 1.

3.2. TPC content using the quick methods

3.2.1. Testo 265

The TPC results obtained using the Testo 265 showed a positive linear correlation with the results obtained using the conventional open-column method (Fig. 1). The correlation equation was $y = 0.89x + 4.0$, with a correlation coefficient of $r = 0.90$, explaining 80.5% of the variation in the results of this quick test. As a result of the fit of the curve and the distribution of the points along the straight line, 95% of the confidence bands and 95% of the prediction bands were very close to a straight line. These results corroborated those of Sánchez-Gimeno and co-workers (Sánchez-Gimeno, Benito, et al., 2008; Sánchez-Gimeno, Negueruela, et al., 2008),

Table 1

Ranges of polar compounds (TPC) in samples of used frying oils.

| Sample | % TPC (Conv. method) | % TPC (Testo 265) | % TPC > 25% ^a (Viscofrit) | % TPC (Fri-check) |
|--------|-------------------------|----------------------|---|----------------------|
| 1 | 9.1 ± 1.6 | 11.8 ± 1.1 | NO/NO | 3.4 ± 0.2 |
| 2 | 19.9 ± 1.4 | 34.8 ± 2.5 | NO/NO | 3.0 ± 0.8 |
| 3 | 22.2 ± 2.2 | 23.5 ± 0.0 | NO/NO | 4.5 ± 1.4 |
| 4 | 10.1 ± 3.6 | 10.8 ± 2.5 | NO/NO | 4.3 ± 0.1 |
| 5 | 11.0 ± 1.5 | 10.8 ± 1.1 | NO/NO | 3.3 ± 0.8 |
| 6 | 14.1 ± 1.0 | 10.8 ± 0.4 | NO/NO | 9.8 ± 0.3 |
| 7 | 11.2 ± 0.1 | 16.8 ± 0.4 | NO/NO | 7.4 ± 0.2 |
| 8 | 32.0 ± 0.9 | 25.8 ± 0.4 | YES/YES | 15.8 ± 0.9 |
| 9 | 35.1 ± 0.7 | 33.5 ± 1.4 | NO/NO ⁺ | 6.1 ± 0.3 |
| 10 | 30.5 ± 0.2 | 27.8 ± 0.4 | NO/NO ⁺ | 3.9 ± 0.1 |
| 11 | 35.5 ± 2.3 | 33.8 ± 0.4 | NO/NO ⁺ | 3.6 ± 0.4 |
| 12 | 14.3 ± 0.2 | 17.0 ± 0.0 | NO/NO | 4.4 ± 0.9 |
| 13 | 8.9 ± 0.0 | 10.3 ± 0.4 | NO/NO | 4.2 ± 0.6 |
| 14 | 14.6 ± 0.3 | 15.8 ± 1.1 | NO/NO | 2.7 ± 0.1 |
| 15 | 8.6 ± 0.7 | 10.8 ± 0.4 | NO/NO | 3.2 ± 0.2 |
| 16 | 10.3 ± 0.8 | 9.3 ± 0.4 | NO/NO | 4.8 ± 0.6 |
| 17 | 7.3 ± 0.0 | 9.0 ± 0.0 | NO/NO | 5.5 ± 0.3 |
| 18 | 6.7 ± 0.6 | 9.3 ± 0.4 | YES/YES ^{**} | 4.5 ± 0.0 |
| 19 | 12.6 ± 0.2 | 15.5 ± 0.0 | NO/NO | 4.0 ± 0.1 |
| 20 | 21.7 ± 3.2 | 17.5 ± 0.7 | NO/NO | 5.8 ± 0.1 |
| 21 | 17.0 ± 1.3 | 13.5 ± 1.4 | YES/YES ^{**} | 5.9 ± 1.8 |
| 22 | 11.5 ± 0.1 | 9.5 ± 0.0 | YES/YES ^{**} | 5.9 ± 2.1 |
| 23 | 30.1 ± 0.5 | 29.8 ± 0.4 | NO/NO ⁺ | 7.0 ± 0.4 |
| 24 | 24.2 ± 0.9 | 24.8 ± 0.4 | NO/NO | 4.4 ± 0.1 |
| 25 | 37.0 ± 0.8 | 32.0 ± 0.0 | NO/NO ⁺ | 10.9 ± 1.2 |
| 26 | 33.0 ± 0.2 | 30.5 ± 0.0 | YES/YES | 4.1 ± 0.1 |
| 27 | 27.4 ± 0.6 | 23.8 ± 0.4 | NO/NO ⁺ | 7.6 ± 2.6 |
| 28 | 32.9 ± 0.3 | 32.0 ± 0.7 | NO/NO ⁺ | 7.8 ± 2.1 |
| 29 | 6.0 ± 0.0 | 9.8 ± 0.4 | NO/NO | 2.4 ± 0.1 |
| 30 | 11.1 ± 2.2 | 7.3 ± 1.8 | NO/NO | 2.6 ± 0.4 |
| 31 | 14.8 ± 0.7 | 17.0 ± 0.7 | NO/NO | 7.2 ± 1.5 |
| 32 | 18.5 ± 0.0 | 12.5 ± 0.0 | NO/NO | 4.2 ± 1.3 |
| 33 | 8.1 ± 0.4 | 14.3 ± 0.4 | NO/NO | 0.0 ± 0.0 |
| 34 | 14.5 ± 0.3 | 19.0 ± 0.0 | NO/NO | 2.4 ± 0.1 |
| 35 | 17.8 ± 2.3 | 22.3 ± 0.4 | NO/NO | 3.3 ± 0.9 |
| 36 | 20.2 ± 0.9 | 25.5 ± 0.0 | NO/NO | 7.0 ± 1.0 |
| 37 | 24.4 ± 1.7 | 28.0 ± 0.0 | YES/YES ^{**} | 12.2 ± 1.3 |
| 38 | 10.7 ± 1.0 | 16.3 ± 0.4 | NO/NO | 0.0 ± 0.0 |
| 39 | 22.9 ± 0.4 | 26.5 ± 0.0 | NO/NO | 5.4 ± 1.8 |
| 40 | 28.2 ± 0.2 | 34.5 ± 0.0 | YES/YES | 17.1 ± 1.0 |
| 41 | 33.8 ± 0.2 | 42.3 ± 1.1 | YES/YES | 29.2 ± 1.5 |
| 42 | 39.2 ± 1.0 | 41.8 ± 0.4 | YES/YES | 35.2 ± 5.7 |
| 43 | 6.0 ± 1.1 | 17.0 ± 0.7 | NO/NO | 1.2 ± 1.6 |
| 44 | 23.7 ± 1.4 | 28.0 ± 0.0 | NO ⁺ /NO | 7.5 ± 0.1 |
| 45 | 28.7 ± 0.5 | 35.0 ± 0.7 | YES/YES | 26.5 ± 0.7 |
| 46 | 35.2 ± 0.3 | 39.0 ± 0.7 | YES/YES | 27.7 ± 1.1 |
| 47 | 7.6 ± 0.4 | 11.8 ± 0.4 | NO/NO | 4.3 ± 0.1 |
| 48 | 16.1 ± 1.0 | 19.8 ± 0.4 | NO/NO | 9.6 ± 1.4 |
| 49 | 18.9 ± 0.2 | 25.3 ± 0.4 | NO/NO | 18.3 ± 1.5 |
| 50 | 27.3 ± 0.2 | 31.3 ± 0.4 | YES/YES | 27.7 ± 1.1 |
| 51 | 6.2 ± 0.8 | 12.8 ± 0.4 | NO/NO | 3.0 ± 1.0 |
| 52 | 14.9 ± 0.3 | 19.5 ± 0.7 | NO/NO | 4.9 ± 0.3 |
| 53 | 20.3 ± 0.3 | 25.3 ± 0.4 | YES/YES ⁺ | 7.0 ± 1.3 |
| 54 | 24.3 ± 0.4 | 27.8 ± 0.4 | YES/YES ⁺ | 15.8 ± 2.1 |
| 55 | 7.0 ± 0.0 | 10.8 ± 1.1 | NO/NO | 3.7 ± 1.5 |
| 56 | 14.4 ± 0.8 | 11.8 ± 0.4 | NO/NO | 4.6 ± 0.1 |
| 57 | 22.9 ± 0.2 | 16.8 ± 1.1 | NO/NO | 5.6 ± 0.1 |
| 58 | 8.2 ± 1.9 | 12.3 ± 0.4 | NO/NO | 4.6 ± 1.3 |
| 59 | 11.9 ± 1.1 | 13.5 ± 0.7 | NO/NO | 4.8 ± 0.3 |

YES = Yes, the TPC content is superior to 25% and the oil should be discarded.

NO = No, the TPC content is inferior to 25% and the oil may be continuously used.

⁺False negative results of Viscofrit.

^{**}False positive results of Viscofrit.

^a TPC > 25% = is the TPC content superior to 25%?

who found linear correlations between the Testo 265 results and the frying cycle numbers. In addition, the angular and linear coefficients of the correlation equation obtained were similar to those found by Dobarganes (Dobarganes, 2007) in his studies with oils of unknown background ($y = 0.92x + 4.34$; $R^2 = 0.9529$), industrial frying sunflower oil ($y = 0.701x + 4.432$; $R^2 = 0.9508$), and laboratory frying oil ($y = 0.9284x + 0.8316$; $R^2 = 0.9689$) samples.

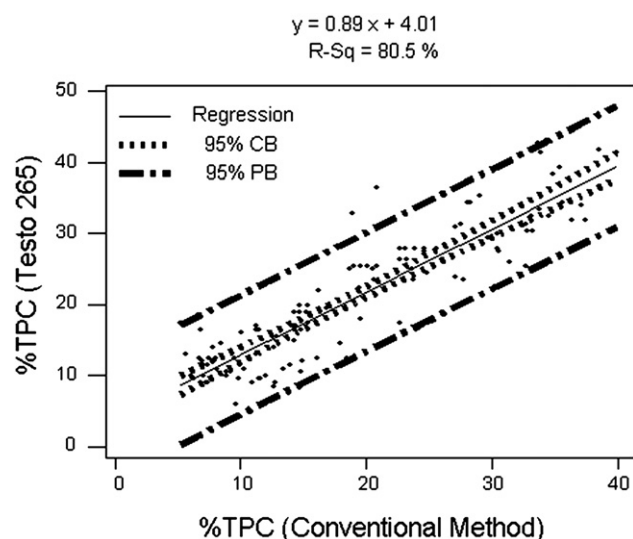


Fig. 1. Correlation graphs for the %TPC provided by Testo 265 and by the conventional method, with 95% of confidence bands (CB) and 95% of prediction bands (PB).

From the means comparison test, no relevant differences were found between the methods at the 5% significance level when the results of the quick test were corrected by a factor of 0.90. For comparative purposes, a correction factor of 0.7 was obtained in a similar study, showing that the results of the Testo 265 could be considered compatible with the results of the conventional method (Juarez, Osawa, Acuña, Sammán, & Gonçalves, 2011). This value for variance can be attributed to a lack of repeatability whenever different equipments are used (Dobarganes, 2007), to the calibration of the equipment or to interference caused by water present in the food. Although Machado and co-workers (Machado, García, & Abrantes, 2007) and Marmesat and co-workers (Marmesat, Rodrigues, Velasco, & Dobarganes, 2007) did not specifically work with Testo 265, they worked with other quick tests whose measuring principle was based on a change in the dielectric constant of the oil after being used for frying. These authors considered the last two factors mentioned as limiting.

The need for correction factors for the Testo 265 results reflects the fact that it overestimates the results. Thus the percent verification was coherent, since there were more false positives than false negatives, with 13.6% (8 samples) and 1.7% (1 sample), respectively, as compared to the 2.5% of false negatives obtained by Dobarganes (Dobarganes, 2007).

The moisture contained in the frying oil is one of the explanations for such behavior, since it increases the dielectric constant (Machado et al., 2007; Marmesat et al., 2007), and as a result, there is an increase in the TPC values. Besides, it must be taken into account that the measurements provided by the Testo 265 are directly connected to the percent of polar compounds in the calibration oil.

3.2.2. Viscofrit

The Signal Test showed that the response obtained with the Viscofrit, aiming to determine whether the frying oil presented more or less than 25% of polar compounds, corresponded to the same response generated by the conventional TPC method. A high total percentage of right answers of 78.8% was obtained, with 9.3% of false positives and 11.9% of false negatives. It is worth considering that 29 of the samples were vegetable fats and 30 were vegetable oils. Of these, 6 vegetable fats and 5 vegetable oils showed false

negatives; whereas the false positives (14) were all attributed to fats.

Marmesat and co-workers (Marmesat et al., 2007) adopted a temperature correction of 22 °C in the analysis of 105 frying oil and fat samples from different origins, and 2 (1.9%) false negatives and 5 (4.8%) false positives were obtained.

In a preliminary study with the Viscofrit, Machado and co-workers (Machado et al., 2007) obtained 22.0% of false negatives and 33.0% of false positives from 33 frying oil samples. Of these, 11 came from frying in hydrogenated fat and the others in palm oil or soybean oil (11 samples each). Due to the high melting point of these samples, all the determinations were estimated for temperatures of 38 °C and the analyses were performed at temperatures of 38 °C (soybean oil), 39 °C (palm oil) and 43 °C (hydrogenated fat).

The use of these temperatures may have favored a higher percentage of false positives and negatives. The Viscofrit requires samples in a totally liquid state and the manufacturer recommends that the temperature used in this quick test be from 20 to 30 °C (Viscofrit, 2006). Although the equipment is capable of working at temperatures between 40 and 50 °C, the difference between the responses for fresh and used oils is greater. Moreover, at high temperatures, the oil quickly cools during the outflow process, even when all necessary care is taken (Antonio Castellón, Viscofrit inventor, personal communications). Thus, the analyses using Viscofrit are more likely to provide less reliable results.

The present study showed that the oil cooled about 2 °C during the test when the initial temperature ranged between 30 and 40 °C. When the range from 40 to 50 °C was considered, the difference could be more than 5 °C, incurring serious experimental errors and influencing the reliability of the results. As an example, quality analyses were carried out on 19 samples using the Viscofrit in two different temperature ranges: 27–39 °C and 41–50 °C. The number of wrong results were 3 (15.8%) and 13 (68.4%), respectively. However, for certain samples, complete melting of the triacylglycerols is not possible at the temperatures recommended by the manufacture.

Certain precautions must be taken with the Viscofrit determinations: the equipment must be clean and calibrated; the sample must be filtered before the analysis, since any food particles may obstruct the Viscofrit hole; the outflow time must be measured right after the temperature is read and the sample, besides being completely liquid, must be shaken in order to avoid any differences in temperature throughout the mass of oil (Marmesat et al., 2007; Viscofrit, 2006).

As for the use of hydrogenated fats, not all of them can be classified as oleic oils according to their fatty acid profile, and some are stearic oils. In this case, the time limit must be increased in the same proportion as the time corresponding to the fresh oleic oil is exceeded. For instance, for a sample at 30 °C the initial time should be 10 s longer than the theoretical time for fresh oleic oil, and the maximum oil outflow time at 43 °C should be about 50 s (Antonio Castellón, inventor of the Viscofrit, personal communication).

3.2.3. Fri-check

As far as the Fri-check is concerned, the results generated did not correlate positively with those obtained using the conventional method. The linear regression model ($y = 0.47x - 1.11$) only explained 35.4% of the variations found in the values found for polar compounds, as obtained using the Fri-check ($r = 0.59$), which is considered insufficient.

Notwithstanding, Fig. 2 shows that the two analytical methods gave different responses for vegetable fats, whereas for vegetable oils, their responses were linearly correlated with the line equation $y = 0.92x - 7.60$, in a model that explained 68.0% of the variation in polar compounds as obtained using the Fri-check ($r = 0.82$).

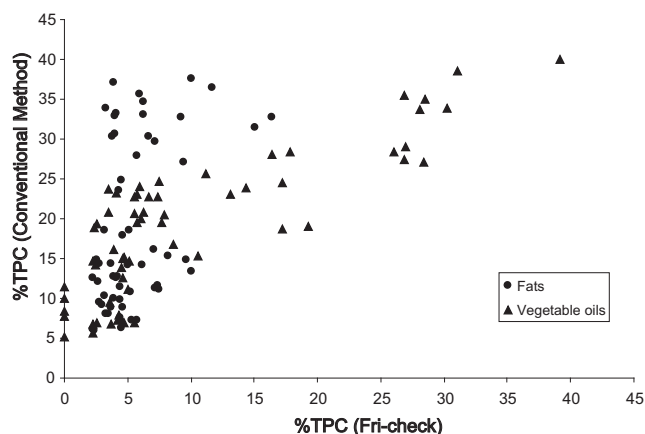


Fig. 2. Correlation graph of %TPC provided by the Fri-check and by the conventional method for vegetable oils and fats.

For TPC values below 10% as obtained using the open-column method, the Fri-check did not offer a good response, since it was not sensitive enough. Thus these values were excluded and a new linear regression carried out with 23 vegetable oil samples. In this case the two methods showed good correlation, which was confirmed by the equation $y = 1.2x - 15.5$, which explained 70.4% ($r = 0.84$) of the variation in the values provided by the Fri-check (Fig. 3).

Nevertheless, the TPC values generated by this quick test were not the same as those obtained using the conventional method, even if correction factors were applied. In this case, correction factors of 1.2; 2.0; 2.1; 2.2; 2.5; 2.9; 3.0 and 4.0 were tested.

Despite the correlation between the two methods, equivalence between them was not possible, probably due to the great dispersion of the points when compared to the linear regression line (Fig. 3), and also because at 95% of confidence, the bands were closer to a hyperbole than to a straight line.

Nothing could be confirmed with respect to whether the kind of oil/fat used or the type of food fried had any influence on the prediction of the values for polar compounds using the Fri-check or the conventional method. However, although the number of samples of the same type of oil (soybean oil, cottonseed oil or palm

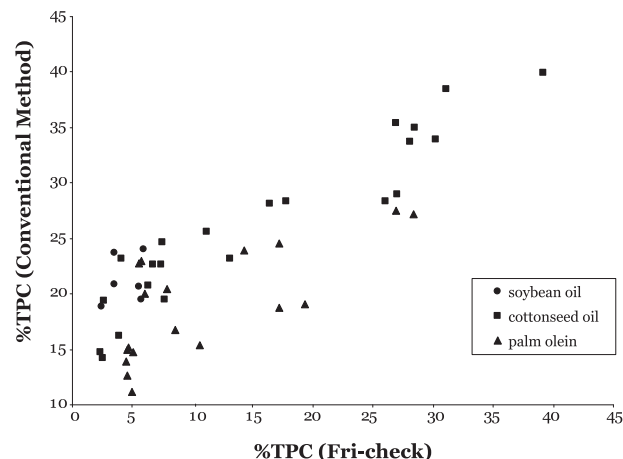


Fig. 4. Correlation graph for %TPC provided by the Fri-check and by the conventional method for soybean oil, cottonseed oil and palm olein samples.

olein) was not significant, Fig. 4 leads one to the conclusion that the kind of oil/fat used or the type of food fried could indeed have had some influence, since the points for the same kind of oil were dispersed and did not follow a linear pattern.

In the preliminary study, with 18 samples of known polar compound contents, a linear correlation of the results provided by the Fri-check was obtained, with the line equation $y = 1.32x - 778$ ($r = 0.97$), and thus the results should be corrected by a factor of 1.25 in order to obtain equivalence with the conventional method (Osawa et al., 2005). Such a correlation can be explained by the fact that the samples had different polar contents, although they were from the same origin and from the same fried food. In this case (the preliminary study), 2 oils ready for disposal were portioned to obtain different polar contents. However, in the present study, samples from different origins and from several fried foods were used, especially in the case of vegetable fat, and so the absence of correlation between the methods could be attributed to this.

Tavares and co-workers (Tavares et al., 2007), in their studies on discarding frying oils in the metropolitan coastal area of the city of Santos, Brazil, evaluated the % TPC using only the Fri-check, and using this procedure the % TPC of some of the samples showed quite high, but unlikely, values, such as 78%. The samples ranged from 4 to 78% for soybean oil and from 9 to 46% for vegetable fats.

Taking everything into account, further studies on possible interferences in the Fri-check test are required.

4. Conclusions

The measurements provided by the Testo 265 presented good correlation with respect to the frying period and the percentages of polar compounds. Of the quick tests studied, this one presented the lowest percentage of false negative results. However, the results were overestimated and correlation factors should be applied in order to evaluate whether the oils should be discarded or not. Calibration of the equipment demands careful attention, and the use of a calibration oil of known origin and content is recommended, so that reading errors can be minimized.

If the experimental procedures are precisely followed, the Viscofrit is a reliable method. For instance, close attention should be paid to the test temperature, filtering of the oil prior to the analyses, and the calibration and cleaning of the funnel. In addition, it is not recommended for use with high melting point samples, due to the low sensitivity of the test at temperatures ranging between 40 and 50 °C.

There was considerable variance between the results of the Fri-check and those of the conventional method for the fat samples.

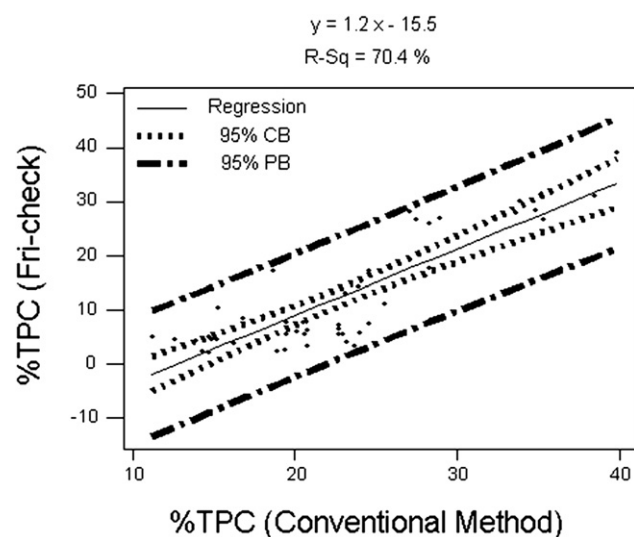


Fig. 3. Correlation graph for %TPC provided by the Fri-check and the conventional method, with 95% of confidence bands (CB) and 95% of prediction bands (PB).

However, the number of frying oil samples examined for the same type of oil and fried food was not sufficient to draw conclusions concerning this quick test. Thus further studies are required with the Fri-check, using a greater diversity of frying oils and fats, as well as different fried foods, so that the action of possible interferences on the test can be evaluated.

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